

09/923,629

Page 1

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L1 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2003 ACS
 ACCESSION NUMBER: 2002:119296 CAPLUS
 DOCUMENT NUMBER: 136:167561
 TITLE: Process for the isolation of sterols from the residues of fatty-acid or methyl-ester production
 INVENTOR(S): Schwarzer, Joerg; Gutsche, Bernhard; Wollmann, Gerhard
 PATENT ASSIGNEE(S): Cognis Deutschland GmbH, Germany
 SOURCE: Eur. Pat. Appl., 7 pp.
 CODEN: EPXXDW
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1179536	A2	20020213	EP 2001-118218	20010728
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, NO				
DE 10038442	A1	20020221	DE 2000-10038442	20000807
US 2002058827	A1	20020516	US 2001-923629	20010807

PRIORITY APPLN. INFO.:

AB A process for obtaining sterols from the residue of fatty acid and/or Me ester prodn. is characterized by: (a) in the residue on hand free fatty acids are esterified with a polyhydroxy or lower monohydroxy alc., after that (b) the mixt. contg. partial glycerides is alcoholized at 90 - 145.degree. and a pressure of 2 - 10 bar over 2 - 20 mins with a lower alc. in the presence of a basic catalyst, (c) after the alcoholysis the excess lower alc. is distilled from the reaction mixt., (d) the alcoholysis catalyst as well as the included glycerin if necessary are sepd., (e) the fatty acid ester is distd. from the mixt. and (f) the bottoms contg. sterol and remaining partial glycerides through a further alcoholysis at 115 - 145.degree. and a pressure of 2 - 10 bars over 4 - 8 h leads to free sterol esters and fatty acid esters. Thus, the distn. residue from the cleavage of soybean oil is treated with glycerin in the presence of tin isooctanoate at 215.degree. and 7 mbar; the residue is then treated with MeOH contg. NaOMe at 137.degree. and 6 bar for 8 mins.; the Me esters are then distd. out; then residue is again treated with MeOH contg. NaOMe for 8 h at 120.degree. and 5 bar; the methanol is then flash evapd. and the catalyst neutralized with citric acid; the product mixt. is washed with H2O to give a product contg. 7.5% free sterols and 0.04% bound sterols; the sterol mixt. contains: 1.2% cholesterol, 1.8% brassicasterol, 23.1% campesterol, 15.3% stigmasterol, 48.9% .beta.-sitosterol, 2.2% .DELTA.5-avenasterol, 0.3% .DELTA.7-avenasterol and 0.05% citrostadienol.

TI Process for the isolation of sterols from the residues of fatty-acid or methyl-ester production

AB A process for obtaining sterols from the residue of fatty acid and/or Me ester prodn. is characterized by: (a) in the residue on hand free fatty acids are esterified with a polyhydroxy or lower monohydroxy alc., after that (b) the mixt. contg. partial glycerides is alcoholized at 90 - 145.degree. and a pressure of 2 - 10 bar over 2 - 20 mins with a lower alc. in the presence of a basic catalyst, (c) after the alcoholysis the excess lower alc. is distilled from the reaction mixt., (d) the alcoholysis catalyst as well as the included glycerin if necessary are sepd., (e) the fatty acid

L1 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)
 ester is distd. from the mixt. and (f) the bottoms contg. sterol and remaining partial glycerides through a further alcoholysis at 115 - 145.degree. and a pressure of 2 - 10 bars over 4 - 8 h leads to free sterol esters and fatty acid esters. Thus, the distn. residue from the cleavage of soybean oil is treated with glycerin in the presence of tin isooctanoate at 215.degree. and 7 mbar; the residue is then treated with MeOH contg. NaOMe at 137.degree. and 6 bar for 8 mins.; the Me esters are then distd. out; then residue is again treated with MeOH contg. NaOMe for 8 h at 120.degree. and 5 bar; the methanol is then flash evapd. and the catalyst neutralized with citric acid; the product mixt. is washed with H2O to give a product contg. 7.5% free sterols and 0.04% bound sterols; the sterol mixt. contains: 1.2% cholesterol, 1.8% brassicasterol, 23.1% campesterol, 15.3% stigmasterol, 48.9% .beta.-sitosterol, 2.2% .DELTA.5-avenasterol, 0.3% .DELTA.7-avenasterol and 0.05% citrostadienol.

ST sterol isolation prodn fatty acid methyl ester; glyceride prepn alcoholysis

IT Fatty acids, preparation
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)
 (esters); process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)

IT Alcoholysis
 Alcoholysis catalysts
 Crystallization
 Distillation
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)

IT Fatty acids, preparation
 Sterols
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)

IT Alcohols, reactions
 Coconut oil
 Palm kernel oil
 Palm oil
 Rape oil
 Soybean oil
 Sunflower oil
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)

IT Glycerides, preparation
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)

IT 124-41-4, Sodium methoxide 30323-21-8, Tin isooctanoate
 RL: CAT (Catalyst use); USES (Uses)
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)

IT 57-88-5P, Cholesterol, preparation 83-46-5P, .beta.-sitosterol 83-48-7P, Stigmasterol 474-40-8P, Citrostadienol 474-62-4P, Campesterol 474-67-9P, Brassicasterol 18472-36-1P,

L1 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)
 .DELTA.5-Avenasterol 23290-26-8P, .DELTA.7-Avenasterol
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)

IT 56-81-5, Glycerin, reactions 67-56-1, Methanol, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (process for the isolation of sterols from the residues of fatty acid or Me ester prodn.)

L1 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2003 ACS
 ACCESSION NUMBER: 2002:119296 CAPLUS
 DOCUMENT NUMBER: 136:167560
 TITLE: Process for the isolation of sterols from the residue of fatty acid ester production
 INVENTOR(S): Gutsche, Bernhard; Bonakdar, Mehdi; Wollmann, Gerhard; Schwarzer, Joerg
 PATENT ASSIGNEE(S): Cognis Deutschland GmbH, Germany
 SOURCE: Eur. Pat. Appl., 8 pp.
 CODEN: EPXXDW
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1179535	A1	20020213	EP 2001-118217	20010728
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
DE 10038456	A1	20020221	DE 2000-10038456	20000807
US 2002082434	A1	20020627	US 2001-923626	20010807

PRIORITY APPLN. INFO.:

DE 2000-10038456 A 20000807

AB A process for obtaining sterols from the residue after distn. of alcoholized oils is characterized by: (a) alcoholysis of the mixt. contg. partial glycerides at a temp. of 115 - 145.degree. and a pressure of 2 - 10 over 5 - 20 mins. with a lower alc. contg. a basic catalyst, (b) after alcoholysis the excess lower alc. is distd. from the reaction mixt., (c) the alcoholysis catalyst is sepd. from the remaining glyceride, (d) the fatty acid alkyl ester is distd. from the mixt. and (e) the bottoms contg. sterol ester and residual partial glyceride through a further alcoholysis at 90 - 145.degree. and a pressure of 2 - 10 bar over 4 - 8 h leads to free sterol and fatty acid ester. Thus, the residue from the distn. of palm kernel oil was treated with MeOH contg. NaOMe at 122.degree. and 5 bar; after 8 min. the catalyst is neutralized with aq. citric acid; the Me ester is distd. at 180.degree. and 3 mbar; the bottoms are treated with more NaOMe in MeOH at 120.degree. for 5 h.

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

TI Process for the isolation of sterols from the residue of fatty acid ester production

AB A process for obtaining sterols from the residue after distn. of alcoholized oils is characterized by: (a) alcoholysis of the mixt. contg. partial glycerides at a temp. of 115 - 145.degree. and a pressure of 2 - 10 over 5 - 20 mins. with a lower alc. contg. a basic catalyst, (b) after alcoholysis the excess lower alc. is distd. from the reaction mixt., (c) the alcoholysis catalyst is sepd. from the remaining glyceride, (d) the fatty acid alkyl ester is distd. from the mixt. and (e) the bottoms contg. sterol ester and residual partial glyceride through a further alcoholysis at 90 - 145.degree. and a pressure of 2 - 10 bar over 4 - 8 h leads to free sterol and fatty acid ester. Thus, the residue from the distn. of palm kernel oil was treated with MeOH contg. NaOMe at 122.degree. and 5 bar; after 8 min. the catalyst is neutralized with aq. citric acid; the Me ester is distd. at 180.degree. and 3 mbar; the bottoms are treated with more NaOMe in MeOH at 120.degree. for 5 h.

ST sterol isolation recovery fatty acid ester prodn; palm kernel oil alcoholysis methanol methoxide catalyst; glyceride prepn alcoholysis methanol methoxide catalyst

IT Fatty acids, preparation

L1 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)
 (esters; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Catalysts
 (for alcoholysis; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Palm kernel oil
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for fatty acid ester prodn.; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Alcohols, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (lower, for alcoholysis; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Distillation
 (of fatty acid esters; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Alcoholysis
 (of glycerides; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Crystallization
 (of **sterols**; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Glycerides, preparation
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
 (partial alcoholysis of; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT **Sterols**
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)
 (process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT Fats and glyceridic oils, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (vegetable, alcoholysis of; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT 67-56-1, Methanol, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (alcoholysis by; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

IT 124-41-4, Sodium methoxide
 RL: CAT (Catalyst use); USES (Uses)
 (alcoholysis catalyst; process for the isolation of **sterols** from the residue of fatty acid ester prodn.)

L1 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)
sterol derivs., the free fatty acids present in the mixt. are esterified with an alc. The mixt. is then transesterified with an alc. in the presence of a basic catalyst.
 After the transesterification, the excess lower alc. is distd. off from the reaction mixt. The transesterification catalyst and the glycerol present, if any, are removed and the fatty acid alkyl ester is distd. off from the mixt. Distn. of fatty acid alkyl esters can be accomplished with a packed column in sequence with a wiped film evaporator. The simultaneous recovery of tocopherol and **sterol** is possible. Tocopherols and **sterols** can be sepd. by the crystn. of **sterols** from a blend of org. solvents.

IT Alcohols, reactions
 RL: PEP (Physical, engineering or chemical process); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)
 (recovery of tocopherols)

IT 64-18-6, Formic acid, uses 64-19-7, Acetic acid, uses 67-56-1, Methanol, uses 67-63-0, Isopropanol, uses 67-64-1, Acetone, uses 75-05-8, Acetonitrile, uses 78-93-3, MEK, uses 100-51-6, Benzyl alcohol, uses 108-87-2, Methylcyclohexane 108-88-3, Toluene, uses 109-94-4, Ethyl formate 109-99-9, THF, uses 110-54-3, Hexane, uses 110-82-7, Cyclohexane, uses 111-65-9, Octane, uses 141-78-6, Ethyl acetate, uses 142-82-5, Heptane, uses 1300-21-6, Dichloroethane 25265-68-3, Methyltetrahydrofuran 29222-48-8, Trimethylpentane
 RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)
 (recovery of tocopherols)

L1 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2003 ACS
 ACCESSION NUMBER: 1995:498453 CAPLUS
 DOCUMENT NUMBER: 122:248289
 TITLE: Recovery of tocopherols
 Inventor: Hunt, Tracy K.; Jeromin, Lutz; Johannsbauer, Wilhelm; Gutsche, Bernhard; Jordan, Volkmar; Wogatzki, Herbert
 PATENT ASSIGNEE(S): Henkel Corp., USA
 SOURCE: PCT Int. Appl., 48 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9504731	A1	19950216	WO 1994-US8481	19940801
W: BR, CA, CN, JP, RU, UA				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
CA 2168856	AA	19950216	CA 1994-2168856	19940801
EP 712399	A1	19950522	EP 1994-924502	19940801
EP 712399	B1	20011114		
R: AT, BE, CH, DE, ES, FR, GB, GR, IE, IT, LI, NL, PT, SE				
BR 9407179	A	19960917	BR 1994-7179	19940801
JP 09502701	T2	19970318	JP 1994-506442	19940801
EP 992499	A2	20000412	EP 1999-118354	19940801
EP 992499	A3	20021211		
R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, NL, SE, PT, IE				
EP 992500	A2	20000412	EP 1999-118355	19940801
EP 992500	A3	20000426		
EP 992500	B1	20020213		
R: AT, BE, CH, DE, ES, FR, GB, GR, IT, LI, NL, SE, PT, IE				
AT 208769	E	20011115	AT 1994-924502	19940801
AT 213239	E	20020215	AT 1999-118355	19940801
US 5616735	A	19970401	US 1995-531366	19950920
US 5646311	A	19970708	US 1996-654483	19960528
US 5670669	A	19970923	US 1996-654441	19960528

PRIORITY APPLN. INFO.:
 US 1993-103628 A 19930806
 US 1994-180592 A 19940113
 EP 1994-924502 A3 19940801
 WO 1994-US8481 W 19940801
 US 1995-531366 A3 19950920

AB Starting from a mixt. contg. tocopherol, fats and/or fat derivs., more particularly fatty acids, and optionally **sterol** and/or **sterol** derivs., the free fatty acids present in the mixt. are esterified with an alc. The mixt. is then transesterified with an alc. in the presence of a basic catalyst.
 After the transesterification, the excess lower alc. is distd. off from the reaction mixt. The transesterification catalyst and the glycerol present, if any, are removed and the fatty acid alkyl ester is distd. off from the mixt. Distn. of fatty acid alkyl esters can be accomplished with a packed column in sequence with a wiped film evaporator. The simultaneous recovery of tocopherol and **sterol** is possible. Tocopherols and **sterols** can be sepd. by the crystn. of **sterols** from a blend of org. solvents.

AB Starting from a mixt. contg. tocopherol, fats and/or fat derivs., more particularly fatty acids, and optionally **sterol** and/or

L1 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2003 ACS
 ACCESSION NUMBER: 1994:280367 CAPLUS
 DOCUMENT NUMBER: 120:280367
 TITLE: Separation of tocopherol and **sterols** from mixts. with fats and/or fatty acids.
 Inventor: Jeromin, Lutz; Johannsbauer, Wilhelm; Gutsche, Bernhard; Jordan, Volkmar; Wogatzki, Herbert
 PATENT ASSIGNEE(S): Henkel K.-G.a.A., Germany
 SOURCE: Ger. Offen., 4 pp.
 CODEN: GWXBX
 DOCUMENT TYPE: Patent
 LANGUAGE: German
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 4228476	A1	19940303	DE 1992-4228476	19920827
DE 4228476	C2	20020502		
WO 9405650	A1	19940317	WO 1993-EP2207	19930818
W: BR, CA, JP, US				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
EP 656894	A1	19950614	EP 1993-919091	19930818
EP 656894	B1	19980225		
EP 656894	B2	20020612		
R: AT, BE, CH, DE, DK, ES, FR, GB, IT, LI, NL, SE				
JP 08500598	T2	19960123	JP 1993-506799	19930818
AT 163416	E	19980315	AT 1993-919091	19930818
ES 2112427	T3	19980401	ES 1993-919091	19930818
BR 9306967	A	19990112	BR 1993-6967	19930818
US 5627289	A	19970506	US 1995-387933	19950227

PRIORITY APPLN. INFO.:
 DE 1992-4228476 A 19920827
 WO 1993-EP2207 W 19930818

AB Title mixts., such as soybean oil steam distillate or tall oil, are treated with a lower alc., preferably MeOH, for esterification of free fatty acids, followed by transesterification, using a basic catalyst. The excess lower alc. is distd. off, and the catalyst and glycerol are removed by washing. After removal of the fatty acid alkyl esters by distn., the tocopherols and **sterols** are isolated by known methods. Esterification of the free fatty acids in the 1st stage is preferably carried out in the presence of strongly-acid ion exchangers.

TI Separation of tocopherol and **sterols** from mixts. with fats and/or fatty acids.

AB Title mixts., such as soybean oil steam distillate or tall oil, are treated with a lower alc., preferably MeOH, for esterification of free fatty acids, followed by transesterification, using a basic catalyst. The excess lower alc. is distd. off, and the catalyst and glycerol are removed by washing. After removal of the fatty acid alkyl esters by distn., the tocopherols and **sterols** are isolated by known methods. Esterification of the free fatty acids in the 1st stage is preferably carried out in the presence of strongly-acid ion exchangers.

ST tocopherol **sterol** sepn fat fatty acid; soybean oil distillate

IT Soybean oil
 RL: BIOL (Biological study)
 (steam distillate of, tocopherols and **sterols** sepn. from)

IT Tall oil
 RL: BIOL (Biological study)
 (tocopherols and **sterols** sepn. from)

09/923,629

Page 4

L1 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2003 ACS (Continued)

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FILE 'CAPLUS' ENTERED AT 08:39:14 ON 09 JAN 2003

L1 4 S STEROL? AND BASIC CATALYST AND ALCOHOL